

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:                   §     Serial No:             unassigned  
  §                             (division of application  
  §                             Serial No. 10/000,746)  
For:   METHOD FOR CUTTING SINGLE-   §  
          WALL CARBON NANOTUBES       §     Filed: concurrently herewith  
  §  
  §     Group Art Unit: 2881 (anticipated)  
  §  
Atty Dkt:     11321-P011C1D2       §     Prior Examiner: Jack I. Berman  
  §                             703.308.4849  
  §

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**PRELIMINARY AMENDMENT ACCOMPANYING REQUEST FOR FILING  
DIVISIONAL APPLICATION UNDER 37 C.F.R. § 1.53(b)**

Sir:

This paper accompanies a Request for Filing Divisional Application Under 37 C.F.R. § 1.53(b) and associated filing fee therefor ("the Request"). If the fee payment is missing or insufficient in amount, or if any other fees are determined to be due, the Assistant Commissioner, Commissioner, and/or the Director of the U.S. Patent & Trademark Office is/are hereby authorized to charge any such fees (or credit any overpayment) to Winstead Sechrest & Minick Deposit Account No. 23-2426, referencing matter number 11321-P011C1D2.

## **AMENDMENTS**

### **In the Title**

Please amend the title by replacing the present title with the following:

-- METHOD FOR CUTTING SINGLE-WALL CARBON NANOTUBES--

### **In the Abstract**

Please amend the abstract by replacing the present abstract with the following:

-- This invention relates generally to cutting single-wall carbon nanotubes (SWNT). In one embodiment, the present invention provides for preparation of homogeneous populations of short carbon nanotube molecules by cutting and annealing (reclosing) the nanotube pieces followed by fractionation. The cutting and annealing processes may be carried out on a purified nanotube bucky paper, on felts prior to purification of nanotubes or on any material that contains single-wall nanotubes. In one embodiment, oxidative etching with concentrated nitric acid is employed to cut SWNTs into shorter lengths. The annealed nanotubes may be disbursed in an aqueous detergent solution or an organic solvent for the fractionation. Closed tubes can also be derivatized to facilitate fractionation, for example, by adding solubilizing moieties to the end caps.--

### **In the Specification**

Please amend the specification as noted on page 4, paragraph 11 of the Request by inserting before the first line of the specification the following:

### **--RELATED APPLICATIONS**

This application is a division of co-pending prior application Serial No. 10/000,746, filed on November 30, 2001, which is a continuation of prior application Serial No. 09/242,040 filed on September 13, 1999, which is the 35 U.S.C. § 371 national application of International

Application Number PCT/US97/13896 filed on August 8, 1997, which designated the United States, claiming priority to provisional U.S. patent application Serial Number 60/023,732 filed on August 8, 1996. Each of the foregoing applications is commonly assigned to the assignee of the present invention and is hereby incorporated herein by reference in its entirety.

This application discloses subject matter related to the subject matter of U.S. patent application Serial Number 09/380,545, filed on September 3, 1999 in the name of Richard E. Smalley et al., entitled "Carbon Fibers Formed From Single-Wall Carbon Nanotubes," which application is commonly assigned to the assignee of the present invention and hereby incorporated herein by reference in its entirety.--

#### **In the Claims**

Please amend the claims as follows.

Please cancel claims 1-83 without prejudice or disclaimer to the subject matter thereof.

Please add the following new claims 84-140:

84. (new) A method for untangling single-wall carbon nanotubes comprising:
- a) providing tangled single-wall carbon nanotubes;
  - b) cutting at least a portion of the single-wall carbon nanotubes to un-tangle at least some of the single-wall carbon nanotubes; and
  - c) recovering a material comprising the single-wall carbon nanotubes un-tangled by the cutting step.
85. (new) The method of claim 84 wherein the single wall carbon nanotubes are cut by a method selected from the group consisting of irradiation with ions, oxidative attack, sonication, oxidative etching, electron beam cutting, plasma arc cutting and combinations thereof.
86. (new) The method of claim 84 wherein the single-wall carbon nanotubes in the material have a mean length that is shorter than a mean length of the single-wall carbon nanotubes in the

tangled single-wall carbon nanotubes.

87. (new) The method of claim 84 wherein the material comprises single-wall carbon nanotubes having lengths in the range between about 5 and about 1000 nm.

88. (new) The method of claim 84 wherein the material comprises single-wall carbon nanotubes having lengths in the range between about 50 and 500 nm.

89. (new) The method of claim 84 further comprising forming a suspension of the tangled single-wall carbon nanotubes in a liquid medium.

90. (new) The method of claim 89 wherein the liquid medium comprises a hydrocarbon.

91. (new) The method of claim 89 further comprising refluxing the suspension.

92. (new) The method of claim 89 further comprising sonicating the suspension with acoustic energy.

93. (new) The method of claim 84 wherein the tangled single-wall carbon nanotubes comprise single-wall carbon nanotubes in a form selected from the group consisting of ropes and mats.

94. (new) The method of claim 93 further comprising dispersing the tangled single-wall carbon nanotubes in a medium selected from the group consisting of an aqueous detergent solution and organic solvent.

95. (new) The method of claim 94 wherein the dispersing is by sonication in a liquid selected from the group consisting of benzene, toluene, xylene, naphthalene, 1,2-dichloroethane and combinations thereof.

96. (new) The method of claim 84 wherein the recovery comprises filtration.

97. (new) A method for shortening the lengths of single-wall carbon nanotubes comprising:

- a) providing a material comprising single-wall carbon nanotubes;
- b) cutting at least a portion of the single-wall carbon nanotubes in the material; and
- c) recovering a product in which a mean length of single-wall carbon nanotubes in the product is less than a mean length of the single-wall carbon nanotube in the material.

98. (new) The method of claim 97 wherein the cutting of the single-wall carbon nanotubes is done by a method selected from the group consisting of sonication of a suspension, energetic ion impact, electron beam cutting, use of a plasma arc, oxidative etching, free radical attack, chemical reactions, heat, pressure, refluxing and combinations thereof.

99. (new) The method of claim 98 wherein refluxing is in concentrated HNO<sub>3</sub>.

100. (new) The method of claim 97 wherein side-walls of at least a portion of the single-wall carbon nanotubes in the material have defects.

101. (new) The method of claim 100 wherein the defects are introduced during the manufacture of the single-wall carbon nanotubes.

102. (new) The method of claim 100 wherein the defects are introduced by a technique selected from the group consisting of free radical attack, energetic ion impact, electron beam cutting, heat, pressure, plasma arc and combinations thereof.

103. (new) The method of claim 100 wherein a portion of the single-wall carbon nanotubes having defects are cut by a method selected from the group consisting of oxidation, oxidative etching, heating, sonication, chemical attack and combinations thereof.

104. (new) A method comprising:

- a) providing a plurality of single-wall carbon nanotubes having at least one open end; and
- b) annealing the plurality of single-wall carbon nanotubes, wherein at least a portion of the open-ends of the plurality of single-wall carbon nanotubes are closed.

105. (new) The method of claim 104 wherein the portion of the open-ends of the plurality of single-wall carbon nanotubes are closed with fullerene caps.

106. (new) The method of claim 104 wherein the annealing step is conducted at a temperature of at least about 1200°C.

107. (new) The method of claim 104 wherein the annealing step is conducted in a vacuum.

108. (new) The method of claim 104 wherein the annealing step is conducted in an inert atmosphere.

109. (new) A method of modifying single-wall carbon nanotubes comprising the step of removing a fullerene cap on at least one end of the single-wall carbon nanotubes.

110. (new) The method of claim 109 wherein the single-wall carbon nanotubes are exposed to an oxidative treatment.

111. (new) The method of claim 110 wherein the oxidative treatment comprises a technique selected from the group consisting of oxidative etching, electrochemical oxidative etching and combinations thereof.

112. (new) The method of claim 110 wherein the oxidative treatment comprises the use of a chemical selected from the group consisting of nitric acid, oxygen, carbon dioxide and combinations thereof.

113. (new) The method of claim 110 wherein the oxidative treatment is conducted at a temperature at most about 500°C.

114. (new) A method comprising:

- a) providing single-wall carbon nanotubes selected from the group consisting of separate single-wall carbon nanotubes, a rope of single-wall carbon nanotubes, a bundle of single wall carbon nanotubes and combinations thereof; and

b) coating the single-wall carbon nanotubes with a coating material.

115. (new) The method of claim 114 wherein the coating material is applied as a liquid.

116. (new) The method of claim 114 wherein the coating material is applied as a vapor.

117. (new) The method of claim 114 comprising contacting the single-wall carbon nanotubes with a vapor comprising a precursor, wherein the precursor is operable to produce a coating on the single-wall carbon nanotubes by a chemical reaction.

118. (new) The method of claim 115 wherein the coating material comprises at least one material selected from the group consisting of metals, polyimide, poly(para-xylene), cyanoacrylate, methacrylate, silicon, silicon dioxide and combinations thereof.

119. (new) The method of claim 115 further comprising removing a portion of the coating.

120. (new) The method of claim 115 wherein the coating material coated on the single-wall carbon nanotubes has a substantially uniform thickness.

121. (new) The method of claim 115 wherein the coating material coated on the single-wall carbon nanotubes has a nanometer scale thickness.

122. (new) The method of claim 115 wherein the coating material comprises a polymer.

123. (new) The method of claim 115 wherein the coating material comprises a fluorescent species.

124. (new) The method of claim 115 wherein the coating material is electrically insulating.

125. (new) A method for producing end-derivatized single-wall carbon nanotubes comprising the steps of:

- a) providing a plurality of single-wall carbon nanotubes;
- b) reacting the single-wall carbon nanotubes with a compound that provides at least

one substituent on at least one of the ends of at least a portion of the single-wall carbon nanotubes; and

c) recovering a material comprising the end-derivatized single-wall carbon nanotubes.

126. (new) The method of claim 125 wherein the at least one substituent is selected from the group consisting of alkyl; acyl; aryl; aralkyl; halogen; substituted thiol; unsubstituted thiol; substituted amino; unsubstituted amino; hydroxy; and OR', wherein R' is selected from the group consisting of alkyl, acyl, aryl, aralkyl, halogen, substituted thiol; unsubstituted thiol; substituted amino; unsubstituted amino, a linear carbon chain and a cyclic carbon chain.

127. (new) The method of claim 126 wherein the linear carbon chain, the cyclic carbon chain, or both, are interrupted by at least one heteroatom.

128. (new) The method of claim 126 wherein the linear carbon chain, the cyclic carbon chain, or both, are substituted with at least one moiety selected from the group consisting of =O, =S, hydroxy, aminoalkyl, amino and a peptide of 2-8 amino acids.

129. (new) The method of claim 125 wherein the end-derivatized single-wall carbon nanotubes are soluble.

130. (new) A method for producing endohedrally-modified single-wall carbon nanotubes comprising:

- a) forming single-wall carbon nanotubes;
- b) introducing the endohedral species during the formation; and
- c) recovering a material comprising the endohedrally-modified single-wall carbon nanotubes.

131. (new) The method of claim 130 wherein the endohedral species is selected from the group consisting of fullerene molecules, C<sub>60</sub>, C<sub>70</sub>, metal atoms and combinations thereof.



132. (new) A method for producing endohedrally-modified single-wall carbon nanotubes comprising:

- a) providing a plurality of single-wall carbon nanotubes that are open at at least one end;
- b) introducing at least an atom or molecule into the inside of the plurality of open single-wall carbon nanotubes; and
- c) recovering a material comprising the endohedrally-modified single-wall carbon nanotubes.

133. (new) The method of claim 132 wherein the endohedral species is selected from the group consisting of fullerene molecules, C<sub>60</sub>, C<sub>70</sub>, metal atoms and combinations thereof.

134. (new) A method comprising:

- a) providing a mixture of single-wall carbon nanotubes;
- b) dispersing the single-wall carbon nanotubes; and
- c) fractionating the mixture by a characteristic selected from the group consisting of lengths, diameters, helicities and combinations thereof.

135. (new) The method of claim 134 wherein the dispersing is enabled by derivatization of the single-wall carbon nanotubes.

136. (new) The method of claim 134 where fractionation is facilitated by derivatization of the single-wall carbon nanotubes.

137. (new) The method of claim 134 wherein the fractionation technique is selected from the group consisting of electrophoresis, DNA fractionation procedures, polymer fractionation procedures and combinations thereof.

138. (new) The method of claim 134 wherein the fractionation of single-wall carbon nanotubes is facilitated by a cutting mechanism having a rate that is dependent on the helicity of the single-wall carbon nanotubes.

139. (new) The method of claim 137 wherein the electrophoresis technique fractionates different structure types of single-wall carbon nanotubes having different polarization and electrical properties.

140. (new) The method of claim 136 wherein the derivitization is with a moiety operable to preferentially bond to one type of single-wall carbon nanotube structure.

\* \* \* \* \*

**REMARKS**

1. *Status of the Application.* Claims 1-83 are cancelled herein without prejudice or disclaimer to the subject matter thereof. Claims 84-140 are added herein. No new matter is added by the addition of these claims.

\* \* \* \* \*

It is believed that each of the claims now pending in the present application recites elements neither taught nor suggested by the prior art. Further, it is believed that the application as a whole is in proper form and condition for allowance. If the Examiner believes that the application may be placed in even better condition for allowance, he or she is invited to contact the undersigned at the telephone number noted below. Alternatively, or in addition, if the Examiner believes that an Examiner interview would be beneficial, the Examiner is invited to note that the undersigned has ready access to the videoconferencing facilities of the South Central Intellectual Property Partnership at Rice University in Houston, Texas. The inventors and the undersigned would welcome the opportunity to use those facilities to clarify any issues deemed to remain unresolved.

Respectfully submitted,

Date: 21-DEC-2001

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